Patent
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2881

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE
BEFORE THE BOARD OF APPEALS

IN RE APPLICATION OF:
KUHLMAN

SERIAL NO. : 10/629,015

FILED: JULY 29, 2003

FOR: A METHOD OF SAMPLE PREPARATION FOR ATOM PROBES AND SOURCE OF SPECIMENS

Examiner: K.T. Nguyen

Group Art Unit: 2881

APPEAL BRIEF
37 CFR 1.192

Real Party in Interest

The real party in interest is the assignee of the application, the Trustees of California Institute or Technology.

Related Appeals and Interferences

There are no related appeals or interferences.

Status of Claims

Claims 1 – 24 were original in the application. Claims 25 – 40 were added. Claims 7, 12, 28, 31, 38 and 40 have been cancelled. Claims 1 – 6, 8 – 11, 13 – 27, 29, 30, 32 – 37, 39, 41, and 42 are appealed.

Status of Amendments

An amendment after final was filed, but to date no advisory action was taken on the amendment. The status of the amendment is indeterminate. The Examiner should be directed to enter the amendment after final. The applicant's brief assumes that the amendment after final has not been entered.

Summary of Invention

The invention is a method of preparing specimens 24 of nonelectropolishable materials for analysis by atom probes which is a superior alternative to the prior methods. In comparison with the prior method, the present method involves less processing time. Whereas the prior method yields irregularly shaped and sized specimens 24, the claimed method offers the potential to prepare specimens of regular shape and size. See paragraph [010]. The invention is directed in claim 1 to a mass production method for the preparation of individual specimens for atom probe analysis. One could refer to it as a method for the preparation of a multiplicity of individual specimens for atom probe analysis without any loss of meaning.

The specimens are preferably substantially nonconductive or semiconductive or insulative as stated in claim 25, paragraph [040] or nonmetallic as stated in claim 29.

The process starts with a slab of material 12 in Fig. 1 from which the specimen will be taken or analyzed. A plurality of removable high aspect ratio posts 24 are defined in the slab 12 without the use of lithography as shown in Fig. 2. The posts 24 are removed from the slab 12 as shown in Fig. 3. See paragraphs [045] – [048].

The invention is also directed in claim 19 to a source of specimens 24 for use in atom probe analysis. A slab of material is manufactured to define and carry a plurality of high aspect ratio posts as shown in Figs. 13, 14, 16 and 17 and described at paragraphs [049] - [051].

Issues

1. Are the terms, “mass production”, “mass produced preparation of individual specimens”, “mass produced specimens”, “semiconductive or insulative specimens”, “nonmetallic specimens”, “without the use of any photolithographic step”, and “without the use of lithography” as used in claims 1, 25, 29, 37, 39, 41 and 42 subject matter understood by one with ordinary skill in the art to be in the possession of applicant?
2. Did the Examiner's questions concerning these terms have any relationship to the questions regarding the reasons that the posts are defined without the use of lithography, or how the semiconductive, insulative or nonmetallic probe operated in an atom probe microscope for analyzing a sample?
3. Did the Examiner error in requiring that each limitation or a single word recited in claim must be defined in the specification?
4. Does **Larson** disclose each and every step as combined in the method claimed in claims 1 – 4, 8, 11, 14 – 26, 29, 33, 37 and 39, for example does **Larson** define a plurality of removable posts in the slab without the use of lithography?

5. Is the subject matter claimed in claims 5, 6, 9, 10, 13, 27, 30, 32, 34 – 36, 41 and 42 obvious in view of **Larson** alone, for example is the use of nonlithographic removal techniques for mass fabrication of atom probe specimens motivated by, taught by, or inferable from **Larson** as a mere design choice?
6. Did the Examiner error in his lack of response to applicant's MPEP 2144(c) notice?

Grouping of Claims

The grouping of claims, each group of which do not stand or fall together, are:

1. Claims 1 – 6, 8 – 11, 13 – 27, 29, 30, 32 – 37, 39, 41, and 42 were rejected under 35 USC 112, first paragraph.
2. Claims 1 – 3, 14, 16, 19, 20, 25, 37 and 39 were rejected under 35 USC 102b.
3. Claims 4 and 21 were rejected under 35 USC 102b.
4. Claims 8, 11, 29, and 33 were rejected under 35 USC 102b.
5. Claims 15 and 17 were rejected under 35 USC 102b.
6. Claim 35 was rejected under 35 USC 102b.
7. Claims 18 and 24 were rejected under 35 USC 102b.
8. Claim 23 was rejected under 35 USC 102b.
9. Claims 5, 6, 13, 27, 32, 41, and 42 were rejected under 35 USC 103a.
10. Claims 9, 10 and 30 were rejected under 35 USC 103a.
11. Claim 35 was rejected under 35 USC 103a.
12. Claim 36 was rejected under 35 USC 103a.

Argument

Rejection Under 35 USC 112, First Paragraph

It is error to hold that the specification fails to convey that the applicant was in possession of the invention. The Examiner contended that the terms:

1. "mass production" could not be understood in claim 1.
2. "semiconductive or insulative specimens" could not be understood in claim 25.
3. "mass production", "nonmetallic specimens" and "without the use of any photolithographic step" could not be understood in claim 29.
4. "mass production" and "semiconductive or insulative specimens" could not be understood in claims 37 and 39.
5. "mass produced preparation of individual specimens" and "without the use of lithography" could not be understood in claims 41.
6. "mass produced specimens" could not be understood in claims 42.

Specifically, the Examiner asked:

1. What is mass production?
2. What are the reasons that the posts are defined without the use of lithography?
3. How is the semiconductive, insulative or nonmetallic probe operated in an atom probe microscope for analyzing a sample?

Ignoring that words have meanings based on a commonly understood definitions shared by users of a language, the Examiner erroneously contends that each limitation or a single word recited in claim must be defined in the specification. This contention is in error. It is very clear from the Federal Circuit's opinion in *Phillips v. AWH*

Corporation, et.al., 2005 U.S. App. Lexis 13954 (CAFC July 12, 2005) that the meaning of the words in a claim need not be defined only in the specification, but may draw from dictionaries, treatises, or other sources, which show how the words are used by ordinary practitioners in the art.

The first paragraph of 35 USC 112 is complied with as stated in the remarks in the first amendment as follows. The use of an atom probe microscope the invention is directed to a method for preparing a specimen for atom probe analysis and is not to the operation of itself of atom probe analysis. The Examiner appears to confuse the atom probe microscope with the probes or specimens that are analyzed.

Answering the third question first, *it is well understood in the art to ordinary practitioners* that the specimen to be examined must be formed into a sharp tip. A positive potential is applied to the tip such that a very large electric field is present at the tip. The ambient gas surrounding the tip is usually helium or neon at a pressure of 1-3 x 10^{-3} millibar. The gas atoms move towards the tip and strike it. The gas atoms may strike the surface many times, before an electron from the gas atom tunnels into the metal tip leaving the gas atom positively ionized. The gas atom is then accelerated away from the tip where it strikes a fluorescent screen. The net effect of many gas atoms is to create a pattern on the fluorescent screen showing spots of light which correspond to individual atoms on the tip surface. The technique was invented by Erwin Müller in 1951. The atom probe is a related technique whereby a sudden voltage pulse is applied to the tip. This causes atoms on the surface of the tip to be ejected. The atoms travel down a drift tube where their time of arrival can be measured. The time taken for the atom to arrive at the detector is a measure of the mass of that atom. Thus

compositional analysis of the sample can be carried out on a layer by layer basis. (See http://www.uksaf.org/tech/fim.html)

Thus, when the Examiner first questions how the nonconductive or nonmetallic sample is used in an atom probe microscope to analyze the sample, it can be readily understood that the first step is to appropriately provide the sample with a sharp tip. The invention provides a solution where a large number of appropriate shaped specimens can be economically manufactured for atom probe microscopy.

Second, the Examiner questions what was the method or apparatus for nonphotolithographically defining the posts. The application discloses several methods which do not use photolithography. The preferred embodiment is sawing intersecting grooves into a substrate with a dicing saw to define an array of posts in the substrate and then fracturing the posts from the substrate. Any nonphotolithographic means for removing of the substrate to form intersecting grooves can be employed. Claim 25, for example, is generic to all means. In addition to mechanically sawing the intersecting grooves into the substrate with a dicing saw, there is disclosed electrical means, which includes electrostatic discharge machining, chemical means, which includes electrolytic and acid etching, and laser means, which includes laser ablation. (See application at page 4, lines 14 – 16 or paragraph [014]).

Finally, the phrase "mass produce" is defined in common dictionaries (Merriam-Webster Online Dictionary to mean "to produce in quantity usually by machinery". See http://www.m-w.com/cgi-bin/dictionary. The term "mass production" is the noun phrase

corresponding to the verb phrase, "mass produce", and means the act of producing in quantity usually by machinery. The specification refers to an array or plurality of posts and the production in quantity is illustrated in Figs. 1 – 3, 14 – 17 and described throughout the specification, including paragraphs [019] – [021], [032] – [035], and [045] – [051].

Rejection Under 35 USC 102b

Claims 1 – 3, 14, 16, 19, 20, 25, 37 and 39 are separately patentable for claiming a nonlithographic method or a mass production source which is made by an inherently nonlithographic method.

Claims 4 and 21 are separately patentable for claiming a method of cross cutting or sawing atom probe specimens or a source of atom probe specimens made by a cross cutting or sawing..

Claims 8, 11, 29, and 33 are separately patentable for claiming a method in which atom probe specimens are made by uniformly removing material around each post.

Claims 15 and 17 are separately patentable for claiming a method for making atom probe specimens in sections including more than one post or a source of atom probe specimens which are made in sections including more than one post.

Claim 35 is separately patentable for claiming a method in which the tip shape of the post is further shaped after being made, but while still being affixed to a section of the slab.

Claims 18 and 24 are separately patentable for claiming a method of forming

atom probe specimens which are separated by a distance on the slab beyond which interference in an atom probe microscope will not occur when used as specimens, and a source in which the posts are separated by a distance on the slab beyond which interference in an atom probe microscope will not occur when used as specimens.

Claim 23 is separately patentable for claiming a source of atom probe specimens are formed on a flattened surface on the slab.

Claims 1 – 4, 8, 11, 14 – 26, 29, 33, 37 and 39 were rejected in a single rejection as anticipated by **Larson**. The Examiner cited the prior method for preparation of a specimen for atom probe described in **Larson** admitting that the prior art method lithographically defines a plurality of posts.

The claimed invention is an improvement over **Larsen**, whose earlier work is cited in the prior art section of the application. Obtaining usable substantially nonconductive specimens for atom probe analysis in quantity and quality at an economic price was the problem which **Larsen** failed to solve. **Larsen** obtained specimens in multilayer thin films, namely a sandwich of NiFe/CoFe/Cu. Unfortunately, geologic samples or rocks do not come as metallic layers and the teaching of **Larsen** of how to form atom probe samples from NiFe/CoFe/Cu thin films is unavailing or inoperable for geologic samples.

Claim 1 calls for the use of a definition of high aspect ratio posts without lithography. Claims 25, 37 and 39 call for a substantially nonconductive specimen. **Larsen** does **not** disclose forming specimens using cross sawing, is directed to only conductive specimens and uses lithography to define the posts. It cannot be sustained that **Larsen** discloses each and every element of claims 1, 25, 37, and 39.

In regard to claims 1 – 3, 16, 25, 26, 37 and 39 **Larson** fails to disclose a nonlithographic method or a mass production source which is made by an inherently nonlithographic method.

In regard to claims 4 and 21 **Larson** fails to disclose cross cutting atom probe specimens or a source of atom probe specimens made by a cross cutting.

In regard to claims 8, 11 and 29 **Larson** fails to disclose a method in which atom probe specimens are made by uniformly removing material around each post.

In regard to claims 15 – 20, and 22 - 24 **Larson** fails to disclose a method for making atom probe specimens in sections including more than one post or a source of atom probe specimens which are made in sections including more than one post.

Rejection Under 35 USC 103a

Claims 5, 6, 13, 27, 32, 41, and 42 are separately patentable for claiming a method for making atom probe specimens using cross cutting and a source of atom probe specimens made by sawing, cutting or cross cutting.

Claims 9, 10 and 30 are separately patentable for claiming uniformly removing the material around the post for mass production of atom probe specimens.

Claim 35 is separately patentable for claiming a method of shaping the tips of the post while the remain connected to a section of the slab.

Claim 36 is separately patentable for claiming a method for forming atom probe specimens in sections with more than one post and a source of atom probe specimens in sections with more than one post.

Claims 5, 6, 9, 10, 13, 27, 29, 30, and 32 - 36 were rejected in a single

rejection as obvious over **Larson** which the Examiner contended disclosed all the features except:

1. cutting the grooves with a saw as recited in claims 5, 13, 27, and 32;
2. nonphotolithographically defining a plurality of regularly shaped posts in the slab as recited in claim 29;
3. removing the material by mechanical means as recited in claim 9;
4. removing the material by electrical means as recited in claims 10 and 30; and
5. separating a section having a plurality of posts from the slab as recited in claim 34.

Each of these steps were considered by the Examiner as mere design choices. **Larson** is limited to a method in which lithography can be used on the material, which is not the case with nonmetallic, insulative or nonconductive materials to which the claimed invention is directed. **Larson** states (page 26, left col., lines 6 – 8):

> "Standard optical lithography and reactive ion etching processes were used to pattern the thermally-oxidized Si wafers."

The posts are then created using the Bosch process which is a deep-trench reactive ion plasma etching step which requires the use of lithography. (page 26, left col., lines 12 – 15)

The Examiner asserts that in the claimed combination the use of various material removal means to make atom probes would be obvious, notwithstanding the extremely small size of the sample and the stringent requirement for a sharp tip being formed. It has always been possible to produce samples for atom probe analysis with the required

small size and sharp tip, but the cost and time required to do so in substantially nonconductive materials has been beyond the reach of the prior art. **Larsen** discloses nothing which is useful for nonlithographic mass production of samples for atom probe analysis. **Ishikawa** in Fig. 2 appears to show a beveled grinding wheel which forms shallow pyramidal structures 24, but even here the teaching is expressly limited to conductive materials (see paragraph 0015 at page 9) and the pyramidal structures 24 are never separated from each other. Instead, an extraction electrode is scanned over the array of pyramidal structures 24 in ultra-high vacuum making measurements until a full data set is obtained. (see paragraph 0018) It can be deduced that shallow pyramidal structures 24 cannot be separated from their underlying substrate to provide separable samples. Further, in regard to claim 1 structures 24 are not cut deeply enough to allow them to be fractured from the substrate, which in turn renders their separation from the substrate into individual specimens impossible.

The Examiner argues that since the Applicant's own disclosure states that sawing can be replaced by other means used for micromachining such as electrostatic discharge machining, acid etching, saw cutting, and laser micromachining, that these other means are rendered obvious. This does not follow. The Applicant's own disclosure of the range of equivalency to a claimed step can never be a basis for a section 103 rejection.

Larson is devoid of any motivation, teaching or leading which would induce a practitioner to abandon the Bosch process for defining a plurality of posts for some nonlithographic removal methodology. The possible use of nonlithographic removal methodologies in larger scale applications where the structures made are not high

aspect ratio posts at small sizes typified by atom microscope specimens, which must be separated from the slab, is not suggested as a viable means in **Larson** or for that matter in the uncited **Ishikawa** reference or any other reference. The Examiner improperly uses the applicant's own disclosure with respect to disclosed nonlithographic removal methodologies as a basis of rejection. The fact that trees can be cross cut to saw lumber out of logs does not mean that atom probe specimens can be cross cut in mass from nonconductive slabs.

Although it is unclear, it appears that the Examiner may be rejecting claims 5, 6, 9, 10, 13, 27, 29, 30, and 32 - 36 on the grounds that the subject matter is common knowledge with respect to the methods for mass production of samples for atom probes. Applicant challenged this contention under MPEP 2144(c) as not properly officially noticed or not properly based upon common knowledge in that at the subject size of the samples, the subject properties of the materials at this size, including particularly nonconductive materials, it was believed that samples could only be reliably mass produced for atom probe analysis by using lithographic methods, and generally only for conductive materials, as evidenced by the cited art. The invention has shown this not to be the case. Arguments concerning the obviousness of the inventive solution are entirely reliant on hindsight derived from the proven results of the invention.

The Examiner made no attempt to respond to the MPEP 2144(c) notice.

In regard to claims 5, 6, 13, 27, 32, 41, and 42 **Larson** does not teach, motivate, suggest or lead to the making atom probe specimens using cross cutting and a source of atom probe specimens made by sawing, cutting or cross cutting.

In regard to claims 9, 10 and 30 **Larson** does not teach, motivate, suggest or

lead to uniformly removing the material around the post for mass production of atom probe specimens.

In regard to claim 35 **Larson** does not teach, motivate, suggest or lead to shaping the tips of the post while the remain connected to a section of the slab.

In regard to claim 36 **Larson** does not teach, motivate, suggest or lead to forming atom probe specimens in sections with more than one post and a source of atom probe specimens in sections with more than one post.

Advancement of the claims to issuance is respectfully requested.

The Commissioner is authorized to charge any additional fees pertaining to this case to Deposit Account No. 01-1960.

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July 20, 2005

7/20/2005

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Appendix

1. A mass production method for the preparation of individual specimens for atom probe analysis comprising:

providing a slab of material from which the specimen will be taken or analyzed;

defining a plurality of removable posts in the slab without the use of lithography; and

removing at least one post from the slab.

2. The method of claim 1 further comprising mounting the post on a pin.

3. The method of claim 1 further comprising shaping the post to a tip shape suitable for use in the atom probe.

4. The method of claim 1 where defining a plurality of posts in the slab comprises cross cutting grooves into the slab.

5. The method of claim 4 where cross cutting grooves into the slab comprising cutting intersecting grooves with a saw.

6. The method of claim 5 where cutting intersecting grooves with a saw comprises cutting at least two sets of parallel grooves at an arbitrarily chosen angle to each other.

8. The method of claim 1 where defining a plurality of posts in the slab comprises forming a plurality of regularly shaped posts in the slab by uniformly removing material around each post to isolate each post from each other post in the plurality of posts.

9. The method of claim 8 where uniformly removing material around each post to isolate each post from each other post in the plurality of posts comprises removing the material by mechanical means.

10. The method of claim 8 where uniformly removing material around each post to isolate each post from each other post in the plurality of posts comprises removing the material by electrical means.

11. The method of claim 8 where uniformly removing material around each post to isolate each post from each other post in the plurality of posts comprises removing the material by chemical means.

13. The method of claim 9 where removing the material by mechanical means comprises removing the material with a dicing saw.

14. The method of claim 2 where removing at least one post from the slab comprises fracturing a single post from the slab.

15. The method of claim 2 where removing at least one post from the slab comprises separating a section from the slab which section includes more than one post connected to the section to provide an array of posts.

16. The method of claim 3 where shaping the post to a tip shape suitable for use in the atom probe comprises focus-ion-beam milling the post to a tip shape.

17. The method of claim 1 further comprising shaping each of the posts to a tip shape suitable for use in the atom probe while each post remains connected to the section.

18. The method of claim 1 where defining a plurality of posts comprises shaping each of the posts so that the posts are spaced by a predetermined distance to avoid interference between separate posts when subsequently used in an atom probe.

19. A source of specimens for use in atom probe analysis comprising a slab of material from which the specimen will be taken, which has been defined into a plurality of posts.

20. The source of claim 19 where at least one post is removed from the slab and the post has been shaped to a tip suitable for use in the atom probe.

21. The source of claim 19 where the plurality of posts defined in the slab have been defined by cross cutting grooves into the slab.

22. The source of claim 20 where the shaped post is focus-ion-beam milled to a tip shape.

23. The source of claim 22 where the slab has a flattened surface into which the posts are defined.

24. The source of claim 19 where defining a plurality of posts comprises shaping each of the posts so that the posts are spaced by a predetermined distance to avoid interference between separate posts when subsequently used in an atom probe.

25. A method for the preparation of individual semiconductive or insulative specimens for atom probe analysis comprising:

providing a slab of semiconductive or insulative material from which the specimen will be taken or analyzed;

defining a plurality of regularly shaped posts in the slab, the posts having a substantially quadrilateral cross section and a prismatic longitudinal axis, the posts being defined by grooves formed into the slab to the depth of the post as the starting material for the specimen; and

removing at least one post from the slab.

26. The method of claim 25 further comprising shaping the post to a tip shape suitable for use in the atom probe.

27. The method of claim 25 where defining a plurality of posts in the slab comprises cross sawing grooves into the slab.

29. A mass production method for the preparation of individual nonmetallic specimens for atom probe analysis comprising:

providing a slab of material from which the specimen will be taken or analyzed;

defining a plurality of regularly shaped posts in the slab without the use of any photolithographic step to a depth of the post as the starting material for the specimen by uniformly removing material around each regularly shaped post to isolate each regularly shaped post from each other regularly shaped post in the plurality of regularly shaped posts;

removing at least one regularly shaped post from the slab; and

selectively removing additional material from the regularly shaped post.

30. The method of claim 29 where uniformly removing material around each post to isolate each post from each other post in the plurality of posts comprises removing the material by electrical means.

32. The method of claim 29 where removing the material around each post to isolate each post from each other post in the plurality of posts comprises removing the material with a dicing saw.

33. The method of claim 29 where removing at least one post from the slab comprises fracturing a plurality of posts from the slab to provide separate specimens.

34. The method of claim 29 where removing at least one post from the slab comprises separating a section from the slab which section includes a plurality of posts which remain connected to the section to provide an array of specimens.

35. The method of claim 34 further comprising shaping each of the posts of the array to a tip shape suitable for use in atom probe analysis while each post remains connected to the section.

36. The method of claim 35 where shaping each of posts comprises shaping each of the posts of the array so that the posts are spaced by a predetermined distance to avoid interference between separate posts when subsequently used in atom probe analysis.

37. A mass production source of individual semiconductive or insulative specimens for use in atom probe analysis comprising a slab of material from which the specimen

will be taken into which material microgrooves have been defined to a predetermined depth to define a plurality of quadrilateral posts.

39. A mass production method for the preparation of individual, semiconductive or insulative specimens for atom probe analysis comprising:

providing a slab of semiconductive or insulative material from which the specimen will be taken or analyzed;

defining a plurality of regularly shaped posts in the slab, the posts having a substantially quadrilateral cross section and a prismatic longitudinal axis, the posts being defined by grooves formed into the slab to the depth of the post as the starting material for the specimen; and

removing at least one post from the slab.

41. A method for the mass produced preparation of individual specimens for atom probe analysis comprising:

providing a slab of material from which the specimen will be taken or analyzed;

defining a plurality of posts in the slab without the use of lithography by sawing intersecting microgrooves in the slab to the depth of the post to provide an array of posts, which is separable from the slab by mechanical fracturing;

removing at least one post from the array; and

machining the at least one post using a focused ion beam to produce the individual specimen.

42. A source for mass produced specimens for use in atom probe analysis comprising a slab of material from which the specimen will be taken into which material intersecting microgrooves have been sawed to a predetermined depth to define a plurality of posts, which depth is determined by the separability of the posts from the slab by mechanical fracturing.

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Invention: **A METHOD OF SAMPLE PERPARATION FOR ATOM PROBES AND SOURCE OF SPECIMENS**

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